Alkali Gelatinization of Starches

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Grain starches, chemically or genetically modified grain starches, tuber starches, and some from other botanical sources were treated at room temperature in media containing a constant amount of water and sodiumhydroxide. Appearances and viscosities of the mixtures were noted during a 7-day quiescent period. The point of complete gelatinization of the starch was deducible from the viscosity changes, as well as from visual observations. Although there were some noticeable differences between the starches, an NaOH to starch ratio of 3.5 to 3.8 meq/g should assure complete gelatinization within several hours or less. This alkali : starch ratio is compared to previous ratios expressed in the literature. A slight dependency of the ratio upon the starch concentration is indicated. The birefringence end-point temperature ranges of the starches were determined, and other implications from the experiments with the 21 materials are discussed.

Alkalische Verkleisterung von Stärken. Getreidestärken, chemisch oder genetisch modifizierte Getreidestärken, Wurzelstärken und einige Stärken anderer botanischer Herkunft wurden bei Raumtemperatur in Medien behandelt, die konstante Mengen an Wasser und Natriumhydroxid enthielten. Das Aussehen und die Viskositäten der Mischungen wurden während einer 7tägigen Ruheperiode beobachtet. Der Punkt der vollständigen Verkleisterung der Stärke wurde sowohl durch die Viskositätsänderungen als auch durch visuelle Beobachtungen festgestellt. Obwohl zwischen den Stärken einige beträchtliche Unterschiede bestanden, stellte ein NaOH/Stärke-Verhältnis von 3,5 – 3,8 meq/g die vollständige Verkleisterung innerhalb mehrerer Stunden oder weniger sicher. Dieses Alkali/Stärke-Verhältnis wird mit früheren in der Literatur angegebenen Verhältnissen verglichen. Eine geringe Abhängigkeit dieses Verhältnisses von der Stärkekonzentration ist nachweisbar. Die Temperaturbereiche für den Endpunkt des Verschwindens der Doppelbrechung bei den Stärken wurden bestimmt, und andere Folgerungen aus den Versuchen mit 21 verschiedenen Substanzen werden diskutiert.

1 Introduction

Most chemical reactions with starch are done with granular starch. However, a few are made after the starch has been gelatinized. For particular reactions the homogeneous, gelatinized substrate may offer an advantage over the heterogeneous, granular form. Starch gelatinization can be accomplished by physical means, enzymatic action, or treatment with chemicals. In the uses of alkali solutions, the most considered are those of *Mangels* and *Bailey* [1] and *Leach*, *Schoch* and *Chessman* [2].

While preparing gelatinized starch solutions, I noted that the amount of NaOH per unit of starch at constant total aqueous volume which would completely disrupt and dissolve ordinary, dent corn, starch granules, would not do the same to high-amylose, corn starch. This observation seemed somewhat in conflict with what *Leach* et al. reported-- "the adsorption (of alkali) affinity is therefore not appreciably influenced by the kind of starch, the amount of the linear or amylose fraction, the granule size, or micelle structure." Additionally, they gave a NaOH level of $0.40 - 0.43$ milliequivalent/g of corn starch as being the "critical amount for gelatinization", and that level is the upper terminal of a Freundlich adsorption relationship. Although a microscope was used to follow the swelling of granules, they gave no details on criteria used to determine the degree of gelatinization.

Contrary to *Leach* et al., *Mangels* and *Bailey* did not definitely

state that they maintained total liquid volume control. They described for their wheat starch granules a microscopic appearance change that showed gelatinization was complete at 0.12–0.15-M NaOH; however, judging by an inflection point in the viscosity of their starch media in an Ostwald pipette, gelatinization was complete at 0.18-M. Assuming 50 ml for their system volumes, the molarity levels convert to 6–9 meq NaOH/g starch, or 14 to 21 times more than the *Leach* et al. figures given above.

The disparity in alkali levels does not permit a deduction of levels needed for gelatinization commencement and completion. The example *Leach* et al. gave to illustrate the efficacy of their method seems also insufficient; they stated that whereas a given volume of alkali solution would gelatinize a certain amount of starch, a 200-fold amount of starch would not be gelatinized by a same volume of the alkali solution. The successful volume of alkali represented 125 meq NaOH/g starch. A later report by *Leach* does not clarify the situation much; he indicated that although a 12.5 meq/g starch level resulted in complete gelatinization of a certain weight of starch in a given volume, that level did not gelatinize 20 times as much starch in a same volume [3].

Thus, in making comparisons of starch gelatinizations, *Leach* et al. were comparing tenfold changes in the amount of starch substrate, a seemingly excessive change to be meaningful. The work reported here was carried out to obtain a more definitive evaluation of the amount of NaOH needed to gelatinize various starches and to make observations on other possible pertinent phenomena accompanying gelatinization.

2 Experimental

2.1 Materials

The starches used were commercial or experimental products, described and obtained from sources indicated in Table 1.

2.2 Procedure

Starch materials were weighed in portions that would contain 2.5, 5.0, 7.5 or 10.0 g of dry substance. Each portion was put into its own 50-ml, screw-capped, clear-glass bottle of 35 mm inside diameter. Enough water (to 0.01 ml accuracy) was added so that it, plus the water contained in the starch, plus the water in the subsequently added aqueous sodium hydroxide solution would total 50 g in the final mixture. The mixture was stirred well by hand with a glass rod having a flattened knob or right-angled end to facilitate mixing from bottom to top to evenly disperse the solids. The stirring was continued while a calculated volume (to 0.01 ml accuracy) of standardized 4.5-N NaOH was added from a free-running pipette. Rapid and thorough mixing was accomplished in just a matter of 3 to 5 seconds with the 2.5 and 5.0 g samples, but in several min with some of the 7.5 and 10.0 g samples as is discussed later.

Each jar was capped and kept at room temperature. At 5 min, 3 h, and every 24 h from the time at which alkali addition was started, the viscosity of the mix was measured in the jar with a Brookfield Model LVF, torsion-wire viscometer, using a No. 4 spindle at 6 rpm, (maximum viscosity measurable, 100,000 cp; Brookfield Engineering Laboratories, Stoughton, MA 02072). During the first 3 h, those mixtures that showed little or no viscosity development, and in which solids readily settled out so that an even appearing suspension was not present, were stirred to again mix evenly every 30 min.

The stiffer syrups and soft gels formed were watched for any tendency to ride up the spindle shaft and give a false, unsteady viscometer reading; viscosity readings were taken before ride-up occurred. The spindle was inserted off-center in the contents, half-way between center and jar wall; a crayon mark was made on the jar wall as a guide to prevent insertion at the same point during subsequent measurements. The rigid and nonflowing gels formed a dimple in their surfaces when the spindle was removed, thus presenting additional guidance insuring that the spindle was not reinserted at the same place. The 35-mm contact length of the spindle reached to 15 mm above the bottom of the jars.

During the setting period, the mixtures were observed for changes in physical appearances, color, and clarity. Clarity was determined by the readability through the jar and contents of an ordinary-sized, typed line of print on white paper held against the far wall of the jar. Translucency was determined by the ability of the medium to transmit the light of a 3 v flashlight bulb held similarly. Black and white photographs of the jars (lettered in alphabetic order and arranged, left to right according to increasing alkali content, in 4 rows representing the 4 used starch concentrations) were taken at 6 and 168 h; color print and slide photographs were taken at 168 h. Photomicrographs of solids isolated from some mixtures were also made at 168 h.

The birefringence end-point temperature (BEPT) ranges of the starches were determined with a Kofler hot stage-polarizing microscope apparatus [4].

3 Results and Discussion

The methodology involved was simple and without notable problems. Even starch suspensions were attained by stirring well before alkali addition. Adding alkali by a free flowing pipette assured rapidity of that addition. The alkali dispersed quickly throughout the media, as evidenced in mixtures made with acid-modified starches or hypochlorite-oxidized starches and highest alkali dosage where uniform mixture clearing was accomplished within 5 min. Also, those mixtures made with starches containing a component that changes color upon alkali treatment, such as the acorn starch, turned color almost instantly throughout when the alkali was added. With the 7.5-g samples at low alkali levels and the 10-g samples at all alkali levels, quick gelation hindered stirring to achieve even, rapid mixing; some striation effects due to ungelatinized granules were noted, especially in the 10-g samples. Those striations, if present after 5 min, disappeared completely before 3 h when an alkali level sufficient for gelatinization had been used. If the alkali level was not sufficient, the entire gel maintained an evenly white, cloudy, translucent to opaque appearance.

A typical experiment is illustrated by Figure 1 with 60 fluidity (60 F), acid-modified, ordinary dent corn starch after 168 h of reaction. Jars A, E and I show that no gelatinization had occurred. Some gelatinization had been effected in Jar B, but there was still an appreciable collection of granules as a settled sediment. Yet in Jars F, K and N, wherein starch concentration had been progressively increased but the NaOH/starch ratio had been kept the same as in Jar B, gelatinization was essentially complete. The slight haze increase from Jars F to K to N was due in part to a "retrogradation effect" that increased with either NaOH or starch concentration, as also evidenced in Jars C, D, H and O. The appearance of Jars J and M suggests too that the trend toward more complete gelatinization with increased (up to a point) starch concentra-
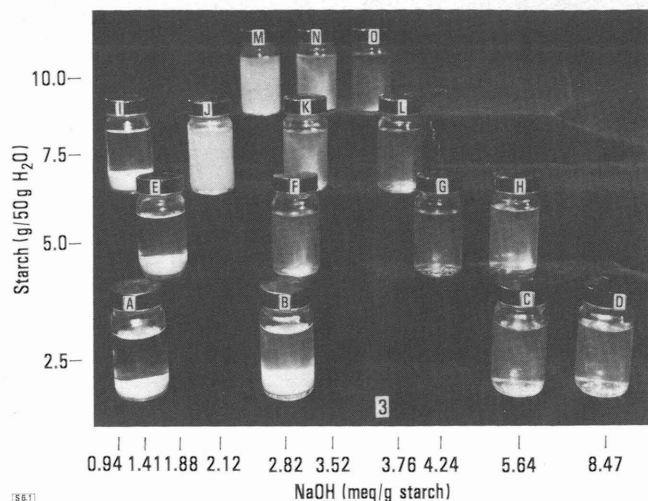
Figure 1. Alkali-gelatinized, acid-modified dent corn starch (60 Fluidity) at varied starch and NaOH concentrations in water. Jar letters correspond to Figure 2 legend letters for concentration level correlations, but not to Figure 2 viscosities. Time, 168 h after alkali addition; temperature, 25°.

tion may be due to impurities in the starch granule that facilitate gelatinization. Others have commented about this phenomenon after using different gelatinization techniques [5 – 7]. Another factor may be that in my system of using a constant amount of total H_2O, an increase in starch amounts results in the "removal" of more H_2O through binding by the starch and hence the effective concentration and reaction of the NaOH is increased in the remaining free H_2O.

Table 1 shows viscosity development data for 21 starches at 4 concentration levels (5, 10, 15, and 20% on wt. starch per wt. water basis) and 4 levels of NaOH during 96 h. From such data one can prepare, as a single graph for each starch, a family of curves for viscosity development with time as exemplified by Figure 2 for Nupana barley starch. Upon comparison of these graphs a number of conclusions can be made, only some of which are commented on as follows.

In those mixtures wherein little, if any, gelatinization had occurred, the viscosity of the supernatant liquid over settled-out starch granules was only slightly higher than that of water; for all of the starches that situation held true at 5% starch concentration and 1.41 meq NaOH/g starch. With some starch types, there was a notable degree of swelling and

Table 1.
Viscosity Development by Starches in Graded-Strength, Aqueous Sodium Hydroxide at 25°C.

Starch Type and Supplier	g Starch/ 50 g H_2O	Meq NaOH/g Starch	Mixture NaOH, N	5 min	3 h	24 h	48 h	72 h	96 h
Corn, dent, wet-milling starch separation product suspension, Grain Processing Corp., Muscatine, IA 52761[1, 2, 3]	2.5	5.64	0.282	12.5	9.5	7.0	5.0	5.0	
	2.5	8.47	0.424	10.0	6.0	4.5	4.4	3.0	
	5.0	2.82	0.282	75.0	60.0	100+	100+	100+	100+
	5.0	4.24	0.424	44.0	35.0	30.5	26.0	21.0	16.3
	5.0	5.64	0.564	41.0	30.5	20.5	16.3	11.5	8.8
	7.5	3.76	0.564	100+	100.0	93.0	86.0	79.5	73.0
	10.0	4.24	0.847	100+	100+	100+	100+	100+	98.0
Corn, dent, pearl starch 3005, CPC International, Englewood Cliffs, NJ 07632[2, 4, 5]	2.5	2.82	0.141	0.3	3.5	9.0	9.8	9.8	9.9
	2.5	5.64	0.282	18.0	12.2	9.0	8.0	7.0	7.0
	2.5	8.47	0.424	15.0	5.0	3.4	2.8	2.7	2.6[6]
	5.0	2.82	0.282	79.0	54.5	100+	100+	100+	
	5.0	4.24	0.424	50.5	38.0	30.0	26.5	21.5	
	5.0	5.64	0.564	37.5	25.5	20.3	16.5	13.5	
	7.5	3.76	0.564	100+	74.5	56.0	44.2	39.0	
	10.0	3.52	0.705	100+	100+	97.5	70.0	60.0	55.0
	10.0	4.24	0.847	100+	89.0	73.5	53.0	43.0	36.0
Corn, dent, pearl starch 3001, CPC International[4, 5, 7]	2.5	2.82	0.141	0.3	0.3	1.8	2.5	2.0	2.0
	2.5	5.64	0.282	4.0	2.5	2.0	1.5[6]	1.5	1.5
	2.5	8.47	0.424	2.0	1.0	0.7	0.7[6]	1.0	1.0
	5.0	2.82	0.282	43.0	25.5	41.0	78.0	85.8	92.0[6]
	5.0	4.24	0.424	19.5	8.7	6.3	5.3	4.2	3.3
	5.0	5.64	0.564	6.5	3.8	2.5	2.4	2.5	2.0
	7.5	1.88	0.282	70.0	100+	100+	100+	100+	100+
	7.5	2.82	0.424	73.5	47.8	100+	100+	100+	100+
	7.5	3.76	0.564	32.5	21.5	14.0	12.5	9.4	7.5
	7.5	4.70	0.705	18.4	12.0	9.2	7.3	5.6	4.5
	10.0	2.82	0.564	63.8	50.2	100+	100+	100+	100+
	10.0	3.52	0.704	45.5	38.8	31.0	26.5	21.5	21.0
	10.0	4.24	0.847	39.3	29.0	20.3	15.0	13.5	12.5
Corn, dent, high-amylose (55 – 60%) starch, Amylon 60, National Starch and Chemical Corp., New York, NY 10017[1]	2.5	5.64	0.282	5.0	2.8	2.0	2.3	2.0	1.5
	2.5	8.47	0.424	2.5	1.5	1.0	1.5	1.5	1.5
	5.0	2.82	0.282	13.5	25.0	24.8	23.5	23.0	22.0
	5.0	4.24	0.424	9.5	4.8	3.5	3.0	2.8	3.0
	5.0	5.64	0.564	2.3	2.0	2.0	2.0	1.8	1.5
	7.5	1.88	0.282	2.3	25.0	22.5	24.3	27.8	27.3
	7.5	2.82	0.424	45.0	38.5	45.0	70.5	95.0	100+
	7.5	3.76	0.564	7.5	7.0	5.0	4.5	3.5	3.0
	10.0	2.12	0.424	74.5	100+	100+	100+	100+	100+
	10.0	2.82	0.564	43.5	47.0	100+	100+	100+	100+
	10.0	3.52	0.705	12.0	11.5	10.0	9.0	8.0	8.0
	10.0	4.24	0.847	5.5	5.0	3.8	3.3	3.0	3.0
Corn, dent, high-amylose (70%) starch, Amylon 70, National Starch and Chemical Corp.[1, 8]	5.0	2.82	0.282	2.5	7.5	7.3	7.3	6.0	5.8
	5.0	4.24	0.424	7.5	4.3	3.3	3.0	2.5	2.8
	5.0	5.64	0.564	2.0	2.0	1.8	1.5	1.3	1.5

Table 1.
Continued

| Starch Type and Supplier | Mixture Composition | | | Brookfield (Model LVF Viscometer No. 4 spindle at 6 r.p.m.) Viscosity, in cp X 10^{-3}, at Various Times After Mixture Preparation | | | | | |
	g Starch/ 50 g H_2O	Meq NaOH/g Starch	Mixture NaOH, N	5 min	3 h	24 h	48 h	72 h	96 h
	7.5	1.88	0.282	1.5	4.8	6.5	6.0	5.8	5.5
	7.5	2.82	0.424	31.5	32.0	27.0	26.8	33.5	48.0
	7.5	3.76	0.564	7.0	4.0	3.5	3.0	2.5	2.5
	10.0	2.12	0.424	35.0	59.0	100+	100+	100+	100+
	10.0	2.82	0.564	35.0	37.0	51.0	89.5	97.0	99.0
	10.0	3.52	0.705	8.0	8.0	6.5	5.5	5.5	5.0
	10.0	4.24	0.847	3.0	3.0	1.5	1.5	1.5	1.5
Corn, waxy, high-amylopectin (100%) starch, Amioca, National Starch and Chemical Corp.[4,5]	2.5	2.82	0.141	1.5	2.0	3.5	5.0	5.0	5.0
	2.5	5.64	0.282	15.0[9]	10.0	8.0	7.0	5.0	4.0
	2.5	8.47	0.424	7.8[9]	4.0	3.0	2.5	2.5	2.0
	5.0	2.82	0.282	65.0	49.0	33.0[6]	37.5	31.0	25.0[9]
	5.0	4.24	0.424	32.0	22.5[6]	21.5	15.0	11.0[9]	6.5
	5.0	5.64	0.564	30.0[6]	15.0	11.0	7.8[9]	5.3	4.0
	7.5	1.88	0.282	96.0	100+	81.0	67.5	71.5	69.0
	7.5	2.82	0.424	61.5	53.5	43.0	29.3	38.3[6]	32.0[6]
	7.5	3.76	0.564	48.0	43.5	28.0	19.5	13.0[6]	8.5[6]
	10.0	2.12	0.424	100+	99.0	96.0	78.5	64.3	50.0[6]
	10.0	2.82	0.564	100+	87.5	72.0	49.5	30.0	20.0[9]
	10.0	3.52	0.704	83.5	67.5	42.0	25.0	16.0[6]	11.0[9]
	10.0	4.24	0.847	75.5	47.5	38.8	20.0	12.5[6]	9.0
Corn, dent, acid-modified (60 fluidity) starch, Clinton 60F, Clinton Corn Proc. Co., Clinton, IA 53732[1,8,10,11]	5.0	2.82	0.282	1.5	2.5	1.5	1.5[9]	1.5	1.5
	7.5	1.88	0.282	1.5	3.5	96.0	100.0	100.0	100.0
	7.5	2.82	0.424	2.0	2.0	2.0	3.0[9]	5.8	7.0
	10.0	2.12	0.424	7.0	100+	100+	100+	100+	100+
	10.0	2.82	0.564	2.5	2.5	42.5	68.0	86.0	93.0
Corn, waxy, high-amylopectin (100%) acid-modified (85 fluidity) starch, Amioca 85, National Starch and Chemical Corp. [8,10,12]	5.0	1.41	0.141	1.5	1.5	2.0	1.5	1.5	1.5
	5.0	2.82	0.282	1.5	1.5	2.0	1.5[9]	1.3	1.5
	7.5	1.88	0.282	1.5	1.3	2.0	1.3	1.3	1.3
	10.0	2.12	0.424	2.0	1.0	1.5	1.3	1.3	1.3
Corn, dent, hydroxyethyl starch, (high fluidity) Penford Gum 290, Penick and Ford, Ltd., Cedar Rapids, IA 52406[8,13,14]	10.0	2.12	0.424	1.0	1.3	2.0	1.3	1.0	1.0
	10.0	2.82	0.564	1.0	1.5	2.0	1.3	1.0	1.0
	10.0	3.52	0.704	1.3	1.8	1.8	1.3	1.0	1.0
	10.0	4.24	0.847	1.3	1.8	1.8	1.5	1.0	1.0
Corn, dent, hypochlorite-oxidized starch, (low fluidity) Stayco S, A. E. Staley Mfg. Co., Decatur, IL 62525[5,8,11,13]	7.5	1.88	0.282	2.3	1.8	1.5	1.5	1.3	1.3
	7.5	2.82	0.424	2.5	1.5[9]	1.3	1.3	1.3	1.3
	7.5	3.76	0.564	2.0[9]	1.5	1.3	1.3	1.3	1.3
	10.0	2.12	0.424	2.5	1.8	1.5	1.5	1.5	1.5
	10.0	2.82	0.564	2.0	1.5[9]	1.5	1.5	1.3	1.3
Acorn (ring-cup oak, *Quercus glauca*, Korea) starch, Wang Brand, Korea-American Food Corp., San Francisco, CA 94124[1,2]	2.5	5.64	0.282	15.0	9.5	4.5	3.0	2.3	2.0
	2.5	8.47	0.424	11.0	5.0	3.3	2.3	2.0	2.0
	5.0	2.82	0.282	73.0	51.5	42.5	67.0	82.5	85.0
	5.0	4.24	0.424	68.0	48.0	29.0	24.5	19.0	17.0
	5.0	5.64	0.564	63.0	34.0	25.0	23.0	18.5	15.5
	7.5	3.76	0.564	100+	100+	79.0	64.0	64.5	59.0
	7.5	4.70	0.705	100+	98.0	69.0	65.0	61.0	53.5
	10.0	3.52	0.705	100+	100+	100+	98.0	100.0	94.0
	10.0	4.24	0.847	100+	100+	100+	95.0	100.0	91.0
Barley (Nupana) starch, Prof. Kenneth J. Goering, Montana State University, Bozeman, MT 59717[2,5]	2.5	2.82	0.141	1.0	9.5	10.0	9.5	10.0	10.0
	2.5	5.64	0.282	11.5	9.0	6.0	3.5	3.0	3.0
	2.5	8.47	0.424	8.5	5.3	2.8	2.5	2.0	1.5
	5.0	1.41	0.141	1.5	2.0	6.5	9.0	14.0	15.5
	5.0	2.82	0.282	84.5	60.0	60.5	90.0	100+	100+
	5.0	4.24	0.424	54.0	46.5	29.0	28.0	20.0	15.0
	5.0	5.64	0.564	43.5	41.0	20.5	16.5	11.0	7.5
	7.5	3.76	0.564	100+	100+	95.0	80.0	55.0	49.0
	10.0	3.52	0.705	100+	100+	100+	100+	100+	89.0
	10.0	4.24	0.847	100+	100+	100+	100+	83.0	82.0
Barley, waxy, high-amylopectin (Campana), large granules only, starch, Prof. Kenneth J. Goering[15]	2.5	2.82	0.141	1.3	11.5	20.0	17.0	15.0	16.0
	5.0	2.82	0.282	80.0	65.0	57.5	45.0	31.3	27.5
	5.0	4.24	0.424	63.5	45.0	42.0	41.0	30.0	19.5
	7.5	1.88	0.282	100+	100+	100+[9]	100+	100+	100+
	7.5	2.82	0.424	100+	100+	96.0	72.0	65.0	58.0
	10.0	2.82	0.564	100+	100+	100+	100+	100+	100+
Bean (mung, *Phaseolus aureus*, Korea) starch, Doug Brand, First Trading Co., Inc., Chicago, IL 60647[2,4,5,15]	2.5	2.82	0.141	1.5	3.5	3.5	4.0	3.0	2.5
	2.5	5.64	0.282	9.5	9.0	4.0	2.3	2.0	1.5
	2.5	8.47	0.424	7.5	5.5	1.8	1.5	1.8	1.5
	5.0	2.82	0.282	75.5	57.0	44.5	55.0	65.5	74.0
	5.0	4.24	0.424	55.0	37.0	14.5	13.0	12.5	9.5
	5.0	5.64	0.564	29.0	29.0	10.0	7.5	5.0	2.5
	7.5	3.76	0.564	100+	98.0	41.0	38.0	37.0	35.0
	7.5	4.70	0.705	100+	90.0	36.0	29.0	25.5	21.0
	10.0	4.24	0.847	100+	100+	75.0	85.0	76.0	95.0

Table 1.
Continued

| Starch Type and Supplier | Mixture Composition | | | Brookfield (Model LVF Viscometer No. 4 spindle at 6 r.p.m.) Viscosity, in cp X 10⁻³, at Various Times After Mixture Preparation | | | | | |
	g Starch/ 50 g H₂O	Meq NaOH/g Starch	Mixture NaOH, N	5 min	3 h	24 h	48 h	72 h	96 h
Potato (Idaho) starch, Boise Cascade Corp., Boise, ID 83728[7]	2.5	1.41	0.070	1.5	2.0	1.5	1.5	1.5	1.5
	2.5	2.82	0.141	25.0	30.5	23.0	26.5	25.5	28.0
	2.5	5.64	0.282	23.5	19.5	14.0	11.5	7.0	5.0
	2.5	8.47	0.424	21.3	18.0	12.0	6.3	2.5	2.5
	5.0	1.41	0.141	6.0	33.0	37.5	48.5	67.0	73.0
	5.0	2.82	0.282	100+	100+	90.0	71.0	62.0	55.0
	5.0	4.24	0.424	100+	65.0	47.5	25.5	15.0	10.0
	5.0	5.64	0.564	100+	50.0	30.0	7.0	3.5	3.0
	7.5	0.94	0.141	3.0	14.0	17.5	15.5	16.0	15.0
	7.5	1.88	0.282	100+	100+	100+	100+	100+	100+
	7.5	2.82	0.424	100+	100+	100+	75.0	60.0	48.5
	7.5	3.76	0.564	100+	100+	77.0	28.5	18.0	14.0
	10.0	2.82	0.564	100+	100+	100+	100+	100+	98.0
	10.0	3.52	0.705	100+	100+	100+	75.0	38.0	40.0
	10.0	4.24	0.847	100+	100+	100+	35.0	16.0	16.0
Rice (Colusa) starch, preparation No. 1919, NRRC, USDA, Peoria, IL 61604[1,15,16]	5.0	2.82	0.282	1.0	1.0	1.0	1.0	1.0	1.0
	7.5	1.88	0.282	1.0	1.0	1.0	1.0	1.0	1.0
	7.5	2.82	0.424	1.0	1.0	1.0[6]	1.0[9]	1.0	1.0
	10.0	2.12	0.424	1.0	1.0	1.0	1.3	1.3	1.3
Rice, waxy, high-amylopectin (Mochi gome) starch, preparation No. 2657, NRRC, USDA, Peoria, IL 61604[1,15,16]	5.0	2.82	0.282	1.0	1.0[6]	1.0[9]	1.0	1.0	1.0
	7.5	1.88	0.282	1.0	1.0	1.0[6]	1.0[9]	1.0	1.0
	7.5	2.82	0.424	1.0	1.0	1.0[9]	1.0	1.0	1.0
	10.0	2.12	0.424	1.0	1.0	1.0[6]	1.0[9]	1.0	1.0
Sorghum (Westland) starch, preparation No. 6365, NRRC, USDA, Peoria, IL 61604[1,15,16]	5.0	2.82	0.282	2.5	1.0	1.0	1.0	1.0	1.0
	7.5	1.88	0.282	2.5	1.5	3.5	15.0	28.0	33.0
	7.5	2.82	0.424	2.0	1.0	1.0	1.0	1.0	1.3
	10.0	2.12	0.424	11.0	7.5	27.0	65.0	88.0	100+
Sorghum, waxy, high-amylopectin (Cody) starch, preparation No. SD-130, NRRC, USDA, Peoria, IL 61604[1,15,16]	5.0	2.82	0.282	5.0	1.5	1.0[6]	1.5[9]	1.0	1.0
	7.5	1.88	0.282	2.0	2.5	1.3	1.5	1.5	1.5
	7.5	2.82	0.424	6.0	2.0	1.5	1.8[6]	1.5	1.5
	10.0	2.12	0.424	17.0	3.5	2.5	2.5	2.0	2.0
Tapioca (Thailand) starch, 0.02% N, 0.11% protein, MM Brand, Eastimpex Agency, San Francisco, CA 94126[1]	2.5	5.64	0.282	2.0	2.0	1.5	1.5	1.5[6]	1.5
	2.5	8.47	0.424	1.5	1.5	1.8	1.5	1.5[6]	1.5
	5.0	2.82	0.282	4.0	2.5	2.0	2.5	2.0	2.0
	5.0	4.24	0.424	4.3	2.0	2.0	2.0	2.0	2.0
	5.0	5.64	0.564	2.5	2.0	2.0	1.5	1.5	1.5
	7.5	1.88	0.282	16.5	19.5	27.5	27.0	27.5	26.5
	7.5	2.82	0.424	7.0	5.0	2.5	2.3	2.5	2.5
	7.5	3.76	0.564	4.5	3.5	2.5	2.0	2.0	2.0
	10.0	2.12	0.424	28.0	16.0	30.5	42.5	53.0	58.5
	10.0	2.82	0.564	13.5	7.5	4.5	3.5	3.0	3.0
	10.0	3.52	0.705	11.0	6.3	3.5	2.5	2.5	2.5
	10.0	4.24	0.847	8.0	5.5	3.5	2.5	2.5	2.5
Wheat, hard red spring, starch, Aytex P. General Mills Inc., Minneapolis, MN 55426[3,5,7]	2.5	2.82	0.141	1.0	4.0	8.5	14.5	16.5	21.0
	2.5	5.64	0.282	5.0	6.0	4.0	3.5	3.0	2.5
	2.5	8.47	0.424	4.5	2.5	2.0	2.0	2.0	2.0
	5.0	1.41	0.141	1.5	3.5	8.5	21.0	38.0	40.0
	5.0	2.82	0.282	43.0	35.5	78.0	100+	100+	100+
	5.0	4.24	0.424	31.0	30.0	20.5	13.3	11.5	9.0
	5.0	5.64	0.564	30.0	28.0	12.0	8.0	6.5	4.5
	7.5	2.82	0.424	80.0	85.0	100+	100+	100+	100+
	7.5	3.76	0.564	67.0	69.0	49.0	34.0	31.0	26.5
	7.5	4.70	0.705	59.0	62.0	25.0	21.0	20.0	16.0
	10.0	4.24	0.847	100+	100+	65.0	45.0	45.0	40.0

[1] At 2.5 g, 1.41 meq NaOH/g and 0.070-N; 2.5 g, 2.82 meq NaOH/g and 0.141-N; 5.0 g, 1.41 meq NaOH/g and 0.141-N; and 7.5 g, 0.94 meq NaOH/g and 0.141-N, no viscosity increase to over 2,000 cp occurs in 96 h.

[2] At 7.5 g, 1.88 meq NaOH/g and 0.282-N; 7.5 g, 2.82 meq NaOH/g and 0.424-N; 10.0 g, 2.12 meq NaOH/g, and 0.424-N; and 10.0 g, 2.82 meq NaOH/g and 0.564-N, the viscosity is 100,000 + cp throughout 96 h.

[3] At 7.5 g, 1.88 meq NaOH/g and 0.282-N; 10.0 g, 2.82 meq NaOH/g and 0.564-N; and 10 g, 3.52 meq NaOH/g and 0.705-N, the viscosity is 100,000 + cp throughout 96 h.

[4] At 2.5 g, 1.41 meq NaOH/g and 0.070-N; and 5.0 g, 1.41 meq NaOH/g and 0.141-N, no viscosity increase to over 1,500 cp occurs in 96 h.

[5] At 2.5 g, 1.41 meq NaOH/g and 0.070-N; and 7.5 g, 0.94 meq NaOH/g and 0.141-N, no viscosity increase to over 2,000 cp occurs in 96 h.

[6] At this point, mixture clarity permits typed line discernment.

[7] At 10.0 g, 2.12 meq NaOH/g and 0.424-N, the viscosity is 100,000 + cp throughout 96 h.

[8] With 2.5 g and all four NaOH strength levels, no viscosity increase to over 1,500 cp occurs in 96 h.

[9] At this point, mixture clarity permits typed line reading.

[10] At 5.0 g, 4.24 meq NaOH/g and 0.424-N; 5.0 g, 5.64 meq NaOH/g and 0.564-N; and 7.5 g, 3.76 meq NaOH/g and 0.564-N, no viscosity increase to over 1,500 cp occurs in 96 h.

[11] With 10.0 g, 3.52 meq NaOH/g and 0.705-N; and 10.0 g, 4.24 meq NaOH/g and 0.847-N, no viscosity increase to over 2,000 occurs in 96 h.

[12] At 7.5 g, 0.94 meq NaOH/g and 0.141-N; 7.5 g, 2.82 meq NaOH/g and 0.424-N; 10.0 g, 2.82 meq NaOH/g and 0.564-N; 10.0 g, 3.52 meq NaOH/g and 0.705-N; and 10.0 g, 4.24 meq NaOH/g and 0.847-N, no viscosity increase to over 1,500 cp occurs in 96 h.

[13] With 5.0 g and all four NaOH strength levels, no viscosity increase to over 1,500 cp occurs in 96 h.

[14] With 7.5 g and all four NaOH strength levels, no viscosity increase to over 1,500 cp occurs in 96 h.

[15] Because of a limited supply of this starch, only the mixtures accounted for were made.

[16] These starches were from the NRRC collection of *MacMasters*. The rice samples were prepared by a water steep process, the sorghums, by a water with SO₂ steep [8].
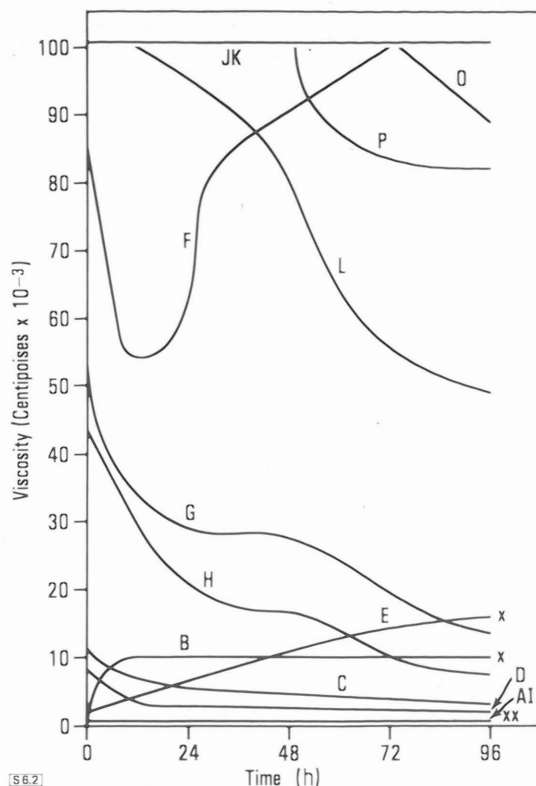
Figure 2. Time versus viscosity development at 25 °C in aqueous mixtures with varied concentrations of barley starch and NaOH.

Curve-plot and concentration legend

Meq NaOH per g Starch	Nupana Barley Starch, g per 50 g H₂O			
	2.5	5.0	7.5	10.0
0.94			I	
1.41	A	E		
1.88			J	
2.82	B	F	K	
3.52				O
3.76			L	
4.24		G		P
5.64	C	H		
8.47	D			

suspension of granules, accompanied by a still insignificant amount of gelatinization but a measurable viscosity increase with time, at concentrations of 5%, with 2.82; 10%, with 1.41; and 15%, with 0.94 (however only potato starch gave any viscosity rise at the last condition). At 10%, with 2.82; 15%, with 1.88; 15%, with 2.82; and 20%, with 2.12 gelatinization was generally considerable and viscosity increase was greatly enhanced, but again varying with starch type. Only the modified and derivatized corn starches, the waxy starches, and the potato, rice, tapioca, and sorghum starches could be judged completely gelatinized at any of those conditions (the sorghum perhaps questionably). Only these starches produced viscosities that increased to a maximum with increasing alkali concentration, up to one of those meq/g points, whereafter further alkali increase resulted in a lower viscosity attainment.

The action of alkali, especially at the higher concentrations, in degrading starch molecule chains to such an extent that they do not contribute much to viscosity may be involved. The various starches may differ in their reactions to alkali. Since air was not excluded, the role of oxygen in color development was noted, especially at the higher starch and alkali levels. Alkaline oxidation might also affect viscosity development.

At 20% starch with 3.52 meq NaOH/g starch, a falling viscosity sometimes was not observed for several days, which indicated a borderline existence of complete gelatinization. However, a handicap to making such a conclusion was the instrument's limitation of 100,000 cp. In such cases visual appearances facilitated decisions. With all starches at all concentrations with 3.76 meq, or more, NaOH/g starch, complete gelatinization was attained in usually several hours or less. This deduction was made from these facts (1) at 2.82, the viscosity direction was upward with time increase, whereas at 3.76, viscosity direction was downward, and (2) in the cases of 15 and 20% starch concentrations, the beginning viscosity points with 3.76, were lower than those with 2.82.

Figure 3 shows some alkali-treated starch photomicrographs that were used along with viscosity data and photographs like Figure 1 to further elucidate the susceptibility of starches to alkali gelatinization. Thus, at 5% starch concentration with 1.41 meq NaOH/g starch, none of the group of Nupana barley, acid-modified (60 F) dent corn, or high-amylose (60%) dent corn starches were gelatinized; but at 5%, with 2.82, the barley starch was completely gelatinized because the photomicrographs did not shown any granules in the translucent syrup obtained. In the cases of 10%, with 1.41 and 15%, with 0.94, the corn starches were essentially ungelatinized, and the barley starch suspension still contained some birefringent granules. However, a photomicrograph after staining with KI − I₂ in 1:1 glycerol water showed that much disorganized starch material encompassed the granules of barley. At 15%, with 1.88, the barley starch produced a translucent gel; the acid-modified corn, an opaque gel; and the high-amylose corn, an opaque syrup that had a heavy concentration of birefringent granules. At these concentrations and dosage levels where gelatinization has been incomplete, the order of decreasing ease of alkali gelatinization was Nupana barley, acid-modified dent corn, and high-amylose dent corn.

Thus, in accord with the viscosity data, the microscopic examinations indicate that more than 2.0 meq NaOH/g starch undoubtedly is needed to assure complete, cold, NaOH gelatinization of starches in general. My finding of the need for at least 2.82 meq NaOH/g of wheat starch at 5% concentration by virtue of viscosity data would convert at my water control level to 0.14-N NaOH, or slightly more; this value is not too far from the 0.18-M *Mangels* and *Bailey* found with 2% wheat starch in water, with the water content perhaps of uncertain control [1].

Table 2 is a summary of the increasing order of gelatinization of the starches by alkali as indicated by the foregoing viscosity and pictorial considerations.

Table 3 lists the decreasing orders of BEPT and amylose contents for the starches. Comparison shows that there is no strict arrangement in position of the starches; but the high-amylose starches occupy the ends representing the most resistance to gelatinization and the high-amylopectin starches, the least resistance. In general, the order for alkali gelatinization agrees better with the order of amylopectin content than with the BEPT. *Goering* et al. likewise found little correlation between viscosity characteristics and the temperature at which 98% of starch granules had lost birefringence, as indicated by the temperature points where Brabender curves left the baseline and recorded a viscosity increase of at least 80 units/min [18]. They believe that the
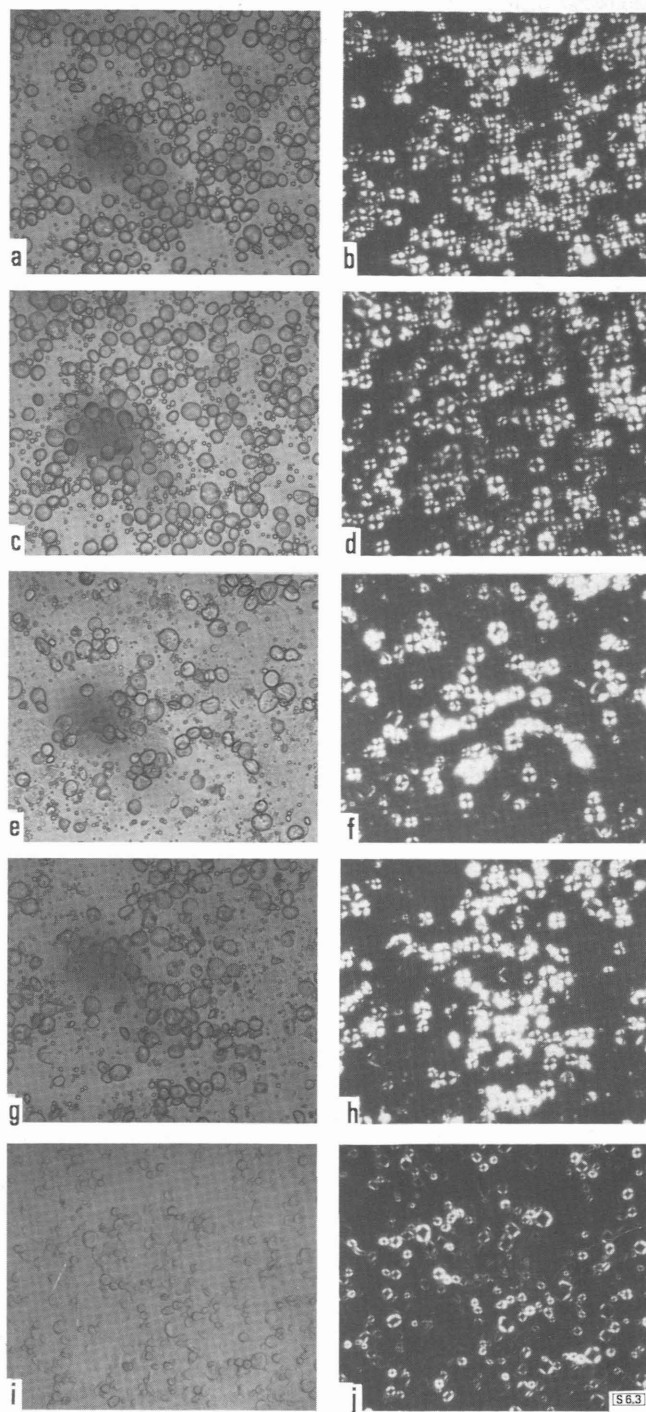
Figure 3. Photomicrographs of alkali-gelatinized starches at varied starch and NaOH concentrations in water at 25°; ordinary light, top row; polarized light, bottom row; magnification 480x.
Starch and starch and NaOH concentrations legend: Nupana barley starch (see Fig. 2 for sampling points); (a) and (b): 2.5 g starch, no NaOH added; (c) and (d): 2.5 g starch, 1.41 meq NaOH/g, Jar A; (e) and (f): 5.0 g starch, 1.41 meq NaOH/g, Jar E; (g) and (h): 7.5 g starch, 0.94 meq NaOH/g, Jar I. High-amylose (60%), dent corn starch; (i) and (j): 7.5 g starch, 1.88 meq NaOH/g, Jar J.

gelatinization temperature should be that to which starch in water must be heated to obtain essentially complete susceptibility of the starch to the enzymic action of glucamylase. However, their order of the % gelatinization determined by the degree of enzymic starch hydrolysis found at the BEPT points for barley, potato, corn, and wheat (in increasing %) does not agree with the positions for the starches in the viscosity-temperature ranking of those authors, in my viscosity ranking order, or in my pictorial (granule appearance) order, but an agreement is closest with the last order named. The factors in correlating starch granule gelatinization to molecular structure, granule structure, birefringence loss temperatures, viscosity development, and enzymic digestion remain complex and somewhat unknown.

At 4.24 meq NaOH/g starch (or 0.68 moles NaOH/mole starch) all of the starches tested were gelatinized at starch concentrations up to 20% in a few minutes. This level is 10 times the critical amount specified by *Leach* et al. and 0.66 to 0.50 times the amount specified by *Mangels* et al., but in the latter case the comparison is compromised because of their lack of total volume control. Actually, if *Leach* et al. meant that by "critical amount for gelatinization" they were signifying complete gelatinization, they may have made a tenfold mathematical error in progressing from their Freund-lich adsorption range of alkali concentrations. To my knowledge they did not publish an *erratum*, but the second report example by *Leach* [3] certainly has a tenfold change in starch amount control.

Although it may be true that different starch types show no differences in regard to their "absorption affinity" for alkali in the "Freundlich range" or pregelatinization stage, I have found that different starches have varied requirements for NaOH for gelatinization and viscosity development; graphs made from data in Table 1 show that no two are the same or superimposable. Even among the first 3 entries, the unmodi-fied dent corn starches, a sensitivity is indicated for the method in distinguishing between closely related starches. Thus, at starch concentrations of 15% and 20%, an evaluation of viscosities produced at the various alkali levels resulted in an increasing gelatinization order of wet corn, pearl 3005, and pearl 3001, which is the same order summarized in Table 2. It is known that the two pearl starches were produced by at least different drying steps which are purported to effect some physical behavorial characteristics of the granules.

Since only a single specimen of each starch type was examined in this work, I cannot say that a sure qualitative or quantitative analytical tool is at hand in this methodology. Refinements might be needed, but as it now stands it seems usable for following amylose content progression over amylopectin in starch granule genetic developments or in telling waxy starches from regular starches, as in corns or barleys. The wide range of starch concentrations used may not be needed to distinguish between starch types. In some cases only the middle range, 10 and 15%, may suffice. In others perhaps one could use only 2 alkali/starch ratio levels at 3 or 4 starch concentrations deemed appropriate. Shoulders and upswings in these viscosity curves may indicate that secondary chemical reactions are involved with alkali. This alkali method needs smaller quantities of starch than a Brabender or Corn Industry Viscometer investigation [19, 20].

Color development by the alkali ranged from pale yellow to yellow to yellow-amber to amber for the starches, the order of increase being: the waxy starches, rice, potato, wheat, dent corn, acid-modified (60 F) dent, mung bean, barley, 60% high-amylose dent, waxy sorghum, hypochlorite-oxidized (low F) dent, 70% high-amylose dent, hydroxyethyl (high F) dent, tapioca, acid-modified (85 F) waxy corn, sorghum, and acorn. The grain and tuber starches, containing less than 0.1% N or 0.5% protein, produced color slowly and lightly, whereas sorghum and acorn starches gave rapid, strong color

Table 2.
Increasing Order[1] of Swelling and Gelatinization of Various Starches in Aqueous Sodium Hydroxide of Varied Concentrations.

Starch Type	As Judged by the Presence of — Ungelatinized Starch Layers in Jars[2]	Opaque Suspensions in Jars	As Judged by the Viscosity Pattern of the Combination of Lowest Starch and NaOH Concentrations that Result in Gelatinization — Starch Type	Gelatinization (% starch, meq NaOH/g, jar) Start	Finish
Waxy corn, acid-modified 85 F	ABEIJ		Dent corn, 70% amylose	15, 1.88, J	20, 3.52, O
Dent corn, 70% amylose	ABEI	FJKMN	Dent corn, 60% amylose	15, 1.88, J	20, 3.52, O
Dent corn, 60% amylose	ABEI	FJKM(N?)	Dent corn, acid-modified 60 F	15, 1.88, J	20, 3.52, O
Sorghum (Westland)	ABEI	(F?)JM	Dent corn, wet	15, 1.88, J	15, 3.76, L[3]
Dent corn, acid-modified 60 F	ABEI	JM(N?)	Acorn (Quercus glauca) Korea	15, 1.88, J	20, 3.52, O
Tapioca, Thailand	ABEI	J(FKMN?)	Dent corn, pearl 3005	15, 1.88, J	20, 3.52, O
Dent corn, hypochlorite-oxidized low F	ABEI	J(FM?)	Mung bean, Korea	15, 1.88, J	15, 3.76, L[3]
			Dent corn, pearl 3001	15, 1.88, J	20, 3.52, O
Rice (Colusa)	ABEI	J(M?)	Sorghum (Westland)	15, 1.88, J	15, 2.82, K
Waxy sorghum (Cody)	ABEI	J	Tapioca (Thailand)	15, 1.88, J	10, 2.82, F
Acorn (Quercus glauca) Korea	ABEI	J	Waxy sorghum (Cody)	15, 1.88, J	20, 2.12, M
Waxy rice (Mochi gome)	ABEI		Waxy corn	15, 1.88, J	20, 2.12, M
Dent corn, wet	AEI	BJMN	Waxy corn, acid-modified 85 F	15, 1.88, J	20, 2.12, M
Mung bean, Korea	AEI	BJM(FKN?)	Rice (Colusa)	15, 1.88, J	20, 2.12, M
Dent corn, pearl 3005	AEI	BJM(N?)	Dent corn, hypochlorite-oxidized low F	15, 1.88, J	20, 2.12, M
Dent corn, pearl 3001	AEI	BJM(N?)	Waxy rice (Mochi gome)	15, 1.88, J	20, 2.12, M
Waxy corn	AEI	BJ(M?)	Dent corn, Hydroxyethyl high F	15, 1.88, J	20, 2.12, M
Dent corn, hydroxyethyl high F	AEI	(BFJKM?)	Wheat, hard red spring	10, 1.41, E	15, 3.76, L
Wheat, hard red spring	AI	BEFJM(KN?)	Barley (Nupana)	10, 1.41, E	20, 3.52, O
Barley (Nupana)	AI	E(JK?)	Waxy barley (Campana)	10, 1.41, E	10, 2.82, F
Waxy barley (Campana)	AI	E	Potato, Idaho	15, 0.94, I	5, 2.82, B
Potato, Idaho	A	EI			

[1] Going down in the columns.

[2] Jar letters, corresponding to Figure 1 array, in which definite starch layer appears. Suspended swollen and dispersed granule strata may increase in depth above the starch layer, also going down in the column.

[3] Differentiation between the conditions of jars N and O cannot be made since the viscosities are over 100,000 cp throughout the test. Actually gelatinization may be complete in jar O, but L's contents yield the detectable fall in viscosity.

Table 3.
Decreasing Order[1] of Birefringence End Point Temperatures and Amylose Contents of Various Starches.

Starch Type	This Work	Literature	[ref.]	Starch Type	% A[2]	% AP[2]	[ref.]
Dent corn, 70% amylose		68 – 76 – 92	[9]	Dent corn, 70% amylose	70	30	[9]
Dent corn, 60% amylose		67 – 75 – 90	[9]	Dent corn, 60% amylose	60	40	[9]
Waxy sorghum (Cody)	66.5 – 71 – 76.5	57.5 – 70.5 – 74	[4]	Mung bean	35	65	[10]
Waxy corn, acid-modified, 85 F	68 – 71 – 75			Dent corn	27	73	[11]
Dent corn, acid-modified, 60 F	66.5 – 70.5 – 73	63.5 – 69 – 73.5	[4]	Sorghum	27	73	[11]
Sorghum (Westland)	65 – 70 – 74.5	68 – 73.5 – 78	[4]	Wheat	25	75	[12]
Mung bean, Korea	65 – 70 – 74	63 – 65 – 69	[10]	Barley	22	78	[12]
Waxy corn	64 – 70 – 73	63 – 68 – 72	[4]	Acorn (Quercus robur, Linn.)	20	80	[13]
Dent corn, hypochlorite-oxidized, low F	63 – 69 – 74	55 – 64 – 73	[4]	Potato, Idaho	20	80	[12]
Tapioca, Thailand	65 – 68.5 – 73	62 – 68 – 73	[4]	Rice	17	83	[14]
Dent corn, wet	63 – 68 – 72			Tapioca	17	83	[14]
Dent corn, pearl 3001 & 3005	63 – 66 – 71	62 – 67 – 72	[4]	Waxy barley	0	100	[14]
Acorn (Quercus glauca) Korea	58 – 65 – 69.5			Waxy corn	0	100	[14]
Potato, Idaho	57 – 63.5 – 67	57.5 – 62 – 67.5	[4]	Waxy rice	0	100	[14]
Dent corn, hydroxyethyl, high F	57 – 62 – 66	58.5 – 62 – 67	[4]	Waxy sorghum	0	100	[14]
Barley (Nupana)	58.5 – 63 – 65	51.5 – 57 – 59.5	[4]				
Waxy barley (Campana)	60 – 63 – 64.5						
Waxy rice (Mochi gome)	58 – 61 – 64	? – ? – 63	[15]				
Wheat, hard red spring	56.5 – 60 – 64	58 – 61 – 65	[16]				
Rice (Colusa, short grain)	58 – 61 – 63	? – ? – 63.5	[17]				

[1] Going down in the columns.

[2] A = amylose, AP = amylopectin.

development, the latter because of the tannins bound to that starch [21]. Among the quick-clearing starches the order of decreasing time needed to reach clarity was: hydroxyethyl (high F) dent, waxy corn, waxy sorghum, acid-modified dent, acid-modified waxy corn, rice, waxy rice, hypochlorite-oxidized (low F) dent, and waxy barley.

The order of increasing "retrogradation" was: acid-modified (85 F) waxy corn, the waxy starches, hypochlorite-oxidized (low F) dent, acid-modified (60 F) dent, potato, rice, tapioca, dent corn, sorghum, mung bean, acorn, hydroxyethyl (high F) dent, barley, wheat, and 60% and 70% high-amylose dent. Through the years, the term "retrogradation" has come to be associated with an effect noted after heat gelatinizing starch or mildly liquefying it with acid or enzyme. Recently it was proposed that for heat-gelatinized starch "retrogradation" may be classified as a "normal or low temperature" one or a "high temperature" one, the latter pertaining to "retrogradation" caused by reaction between starch and highly polar compounds present [22]. Perhaps "retrogradation" occurring with the highly polar alkali gelatinization at 25 °C could be considered different too. In *Bechtel's* gelatinization of defatted corn starch by KOH at room temperature over 5 to 24 h and at 0° for up to 6 days, he noted the appearance of many grainy, small $(1-2 \mu$ d$)$ particles in the media after gelatinization was started, and some seemed to reside also in the swollen but not completely ruptured granules [23]. He considered them possibly to be pre-formed and existent in the granule. However, I wonder if the particles may not have been formed by "retrogradation" because he used such high alkali strengths $(50-125$ meq KOH/g starch$)$. *Mangels* and *Bailey* commented on the more pronounced gelatinization effect of KOH at 0.75-N (*Bechtel* used $0.5-1$-N) over NaOH and the more pronounced destructive effect of K^+ over Na^+ on viscosity [1]. I observed that even with the lower alkali strengths I used on the quick-clearing, modified starches, such as the hydroxyethyl, there was a countering, overpowering clouding due to "retrogradation" in just several hours (less than *Bechtel's* times); the extent appeared to increase as starch and alkali concentrations increased.

The method used here also has been applied to the gelatinization of flours, some from the same grain species as these starches, and the results and comparisons are subjects of a companion paper [24].

4 Summary

This study of the alkali (NaOH) gelatinization of 21 starches of varied botanical sources, and commercial modifications thereof, has shown by visual, microscopic, and viscosity observations that, in general, gelatinization may commence at a level of $0.94-1.88$ meq NaOH/g starch, is more pronounced at $2.12-2.82$, but is not complete until $2.82-3.76$ has been reached or surpassed. There may be some slight variation in the ranges of alkali ratio depending on the starch in water concentration over the spread of $5-20\%$. Each starch type shows a distinctive viscosity development pattern over the concentrations used, and the patterns were found to be non-superimposable. The method has potential as a research tool.

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